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# मानक

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IS 11450 (2006): Method for Determination of Airborne Asbestos Fibre Concentration in Work Environment by Light Microscopy (Membrane Filter Method) [CED 53: Cement Matrix Products]



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( पहला पुनरीक्षण )

*Indian Standard*

METHOD FOR DETERMINATION OF  
AIRBORNE ASBESTOS FIBRE CONCENTRATION  
IN WORK ENVIRONMENT BY LIGHT MICROSCOPY  
(MEMBRANE FILTER METHOD)

( *First Revision* )

ICS 13.040.30

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## FOREWORD

This Indian Standard ( First Revision ) was adopted by Bureau of Indian Standards, after the draft finalized by the Cement Matrix Products Sectional Committee had been approved by the Civil Engineering Division Council.

Exposure to asbestos dust can have harmful effects on the health of workers engaged in industries handling asbestos. In view of safety and health, it is essential to determine concentration of airborne asbestos dust in work environment as reliably as possible. This standard covers the method for determination of airborne asbestos fibre concentration in work environment by phase contrast optical microscopy using the membrane filter method in workplace atmospheres as defined by the counting criteria and was first published in 1986.

Experience has shown that this method does not always produce comparable results when used by different laboratories and by different workers mainly due to variation in sampling, preparation of sample, optical counting and other factors. The differences in results of laboratories and counting may be minimized by strict adherence to the procedure specified in this standard. Membrane filter method is being widely used for more than two decades and has been recommended by various international bodies like International Labour Organization ( ILO ), Asbestos International Association ( AIA ); National Institute of Occupational Safety and Health, USA ( NIOSH ); International Standards Organization ( ISO ); World Health Organization ( WHO ) — 1997.

The membrane filter method is applicable for routine sampling and sample evaluation necessary to assess personal exposure to fibres and to control their presence in occupational environment. This method cannot identify the composition or characteristics of particular fibre types and its use shall be restricted to workplace atmospheres where the predominant fibre types are inorganic.

This standard has been revised based upon:

- a) International standard ISO 8672 ( 1993 ), Air quality-determination of the number concentration of airborne inorganic fibres by phase contrast optical microscopy — membrane filter method, which has been derived from the following three major sources.
  - 1) The rationalization of the many variants used in the asbestos industry, discussed at the Cannes conference and exemplified in the Asbestos International Association's publication.
  - 2) The many experiments carried out in British Laboratories when setting up their central reference scheme.
  - 3) The experiments carried out in European and Canadian Laboratories as well as in one U.S. Laboratory in 1981-82, under the sponsorship of the Canadian European Communities Metals and Minerals Working Group.
- b) AIA Health and Safety Publication, Recommended Technical Method No. 1 ( RTM1 ) reference method for the determination of airborne asbestos fibre concentrations at workplaces by light microscopy. AIA ( UK ).
- c) ASTM Designation D 4240-83 Standard test method for airborne asbestos concentration in workplace atmosphere.
- d) WHO Publication — 1997 Determination of airborne fibre number concentration — A recommended method by phase contrast optical microscopy (membrane filter method).

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( revised )'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## *Indian Standard*

# METHOD FOR DETERMINATION OF AIRBORNE ASBESTOS FIBRE CONCENTRATION IN WORK ENVIRONMENT BY LIGHT MICROSCOPY (MEMBRANE FILTER METHOD)

(*First Revision*)

## 1 SCOPE

1.1 This standard covers the method for determination of number-count concentration of airborne asbestos fibres expressed as the number of fibres/cm<sup>3</sup>.

1.2 This method is applicable for routine sampling and sample evaluation necessary to assess personal exposure to asbestos fibres and their control in occupational environment.

1.3 The use of this method has limitations when applied to samples containing platy or acicular particles and consequently a full qualitative understanding of the sample is necessary to implement this method.

1.4 In this method all particles complying with the defined geometric condition are, in absence of other convincing information, to be considered as asbestos fibres and counted as such, thus ensuring that under-estimates of asbestos exposure are minimized.

## 2 GENERAL METHOD DESCRIPTION

A sample is collected by drawing a measured quantity of air through a membrane filter by means of a battery-powered sampling pump. The filter is later transformed from an opaque membrane into a homogeneous optically transparent specimen. The fibres are then sized and counted using a phase contrast microscope. The result is expressed as fibres per cubic centimetre of air, calculated from the number of fibres on the filter and the measured volume of air sampled.

## 3 SAMPLING APPARATUS AND TECHNIQUE

### 3.1 Filter

Membrane filters (mixed esters of cellulose or cellulose nitrate) of 0.8 to 1.2 µm pore size and a diameter of 25 mm are preferred with printed grids.

### 3.2 Filter Holder

It is necessary to use an open faced filter holder. Due to design of the filter support utilized in some filter

holders, a supporting pad of larger pore size shall be used. The purpose of this supporting pad is to ensure an even distribution of air passing through the primary membrane. Some time it is recommended to use open faced filter holder fitted with a protective metallic cowl.

NOTE — It is generally felt that the use of cowl helps to protect the filter from accidental damage and contamination. But recent evidence suggests that use of cowl is unnecessary, if proper care is taken and use of cowl reduces collection efficiency of the filter because of electrostatic charge it develops due to movement of air.

The internal diameter of the cowl should be at least equal to the exposed diameter of the filter but not more than 2 mm greater than it. Figure 1 shows two possible arrangements.

The cowl helps to protect the filter from accidental contamination. A conducting cowl is preferred to a plastic one because of the possible risk of fibre loss due to electrostatic charge. Filter holders and cowls shall be thoroughly washed before re-use.

Due to the design of the filter support utilized in some filter holders, a supporting pad of larger pore size should be used.

The purpose of this supporting pad is to ensure an even distribution of air passing through the primary membrane.

### 3.3 Storage and Transport

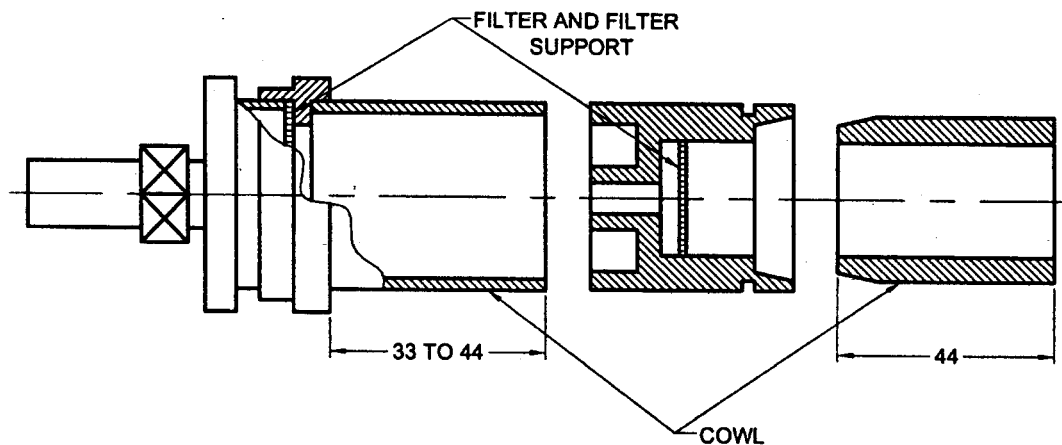
Fixatives shall not be used.

Experience has shown that fixing fibres to the filter surface with cytological or other types of fixatives is unnecessary and this shall not be done.

Filters should be transported in closed holders which should only be opened immediately before use and sealed immediately afterwards.

An alternative is to transfer the filter to a petri dish in the following way:

In a dust-free area, using forceps, carefully remove



All dimensions in millimetres.

FIG. 1 FILTER HOLDERS

each used filter from its holder, taking care to grasp it on its unexposed edge. Place the filter, dust side up, in a plastic petri dish or similar container. Fasten the filter to the bottom of the dish with one or two pieces of adhesive tape attached to the unexposed edge. After transportation, the filter can be removed easily from the dish with a surgical scalpel.

Pack the filter holders or petri dishes into a rigid container with sufficient soft packing material to prevent both crushing and vibration of the filter. Samples shall be unambiguously labelled and caution is necessary to ensure that filters cannot be accidentally re-used. The filters should not be marked for this purpose because of the risk of damaging the filter.

### 3.4 Sampling Pump

A portable battery-operated pump shall be used for personal sampling. The capacity of the battery shall be sufficient to operate continuously over the chosen sampling time. The flow shall be free from pulsation. As a minimum and tentative criterion, there shall be no visible vibration of a variable area flowmeter float when the flowmeter is connected to the filter holder.

Although some pumps are equipped with pulsation dampers, an external damper may have to be installed between the pump and the filter. Never run the pump without a filter.

Connecting tubing shall be constriction-proof and the connections shall be leakproof.

### 3.5 Flowrate

The flowrate shall be adjusted to approximately 1 litre/min (approximately 4 cm/s face velocity). The adjustment of sample density to the range specified

in 3.6 should be done by adjusting sampling time as in 3.8. The flowrate shall be checked at least before and after sampling. If the difference from the initial flowrate is greater than 10 percent, the sample shall be rejected. If an external flowmeter is used to determine the flowrate of the pump, care shall be taken to ensure that the flowmeter does not cause unknown changes to the flowrate. Measurements of the 'sampling train' flowrate using a soap-film flowmeter, with and without the external flowmeter, is one satisfactory method of determining any change in flowrate. The flowmeter used shall be able to measure flowrate to an accuracy within  $\pm 5$  percent of the true flow (95 percent confidence limit) (see Annex A for flowrate calibration).

### 3.6 Acceptable Fibre Loadings on Filters

#### 3.6.1 Minimum Loading

The minimum filter loading should exceed 50 fibres/mm<sup>2</sup> (approximately 0.4 fibres per Walton-Beckett graticule area). In special circumstances (for example, when an indication of concentration with low precision is acceptable), it is permissible to lower the acceptable fibre loading to 20 fibres/mm<sup>2</sup> (approximately 0.15 fibres per Walton-Beckett graticule area).

The lowering of the acceptable fibre loading gives, at best, barely acceptable coefficients of variation.

#### 3.6.2 Maximum Loading

The filter loading should not exceed a maximum of approximately 650 fibres/mm<sup>2</sup> (5 fibres per graticule area averaged for all counted fields) for the majority of sampling situations. This may need to be reduced to an average of about one fibre per graticule area when mixed dusts or agglomerates are present, and can sometimes be doubled when only fibres are present. Average filter loadings exceeding 5 fibres

per graticule area tend to result in an underestimation and should be treated with caution.

### 3.7 Blanks

For each batch of filters used for sampling, and for every 25 filters in the batch, select one unused filter which has been subjected to the same treatment as for normal samples, without having air drawn through it, or having been attached to the worker. If this 'blank' yields fibre counts greater than 3 fibres per 100 graticule areas, the entire sampling and analytical procedure should be examined carefully to find the cause of the contamination.

When the blank count exceeds 3 fibres/100 graticule areas, and also exceeds 10 percent of the actual sample fibre count/100 graticule areas, the samples represented by the blank are not considered acceptable for assessment of worker exposure.

However, the determination may still be useful for indicating compliance with the exposure standard. For example, if the estimated exposure is less than that permitted by regulations even with the contamination, that is, a conservative estimate of compliance.

*Example :*

The fibre count of blank filter was 15 fibres/100 graticule areas (that is, 0.15 fibres/area) while the sample yielded 108 fibres in 90 graticule areas (that is, 1.20 fibres/area).

$$\frac{\text{Blank count}}{\text{Sample count}} (\text{percent}) = \frac{0.15}{1.20} \times 100 = 12.5$$

As this percentage exceeds 10 percent, the sample is rejected. Furthermore, because the blank count exceeded 3 fibres/100 graticule areas, the cause of contamination shall be found and corrected.

### 3.8 Recommended Single Sample Duration

Taking into account the filter loading considerations detailed in 3.6, the duration  $t$ , in min, for each single sample may be determined from the following formula:

$$t = \frac{A}{a} \times \frac{L}{c_{\text{exp}}} \times \frac{1}{r} \quad \dots\dots(1)$$

where

$A$  = effective filter area, in  $\text{mm}^2$ ;

$a$  = graticule area, in  $\text{mm}^2$ ;

$c_{\text{exp}}$  = average fibre concentration, in fibres/ $\text{cm}^3$ , expected to occur during the single sample duration;

$L$  = required filter loading, in fibres per graticule area; and

$r$  = flowrate, in  $\text{cm}^3/\text{min}$ .

To provide guidance on the selection of single sample duration, Table 1 lists recommended single sample durations, based on 2 fibres per graticule area. If it is not possible to use these values, the minimum and maximum durations allow a choice to be made whilst still remaining within the constraints of 3.6. If the concentration is not known and the objective is compliance sampling, the single sampling duration should preferably be that recommended for the appropriate limit.

**Table 1 Single Sample Durations**  
( Clause 3.8 )

Expected Fibre Concentration	Single Sample Duration		
	$t_{\text{Min}}^{1)}$	$t_{\text{recommended}}^{2)}$	$t_{\text{Max}}^{3)}$
Fibres/ $\text{cm}^3$			
(1)	(2)	(3)	(4)
0.1	3.3 h	Full shift	Full shift
0.5	40 min	3 h	8 h
1	20 min	1.5 h	4 h
2	10 min	45 min	2 h
5	4)	20 min	1 h
10	4)	10 min	30 min
20	4)	10 min	10 min

#### NOTES

- 1 Sampling time shall be measured within  $\pm 2.5$  percent.
- 2 The timers or counters, if installed in some pumps, should be checked for reliability.

<sup>1)</sup> 0.4 fibres/graticule area equivalent to 50 fibres/ $\text{mm}^2$ .

<sup>2)</sup> 2 fibres/graticule area.

<sup>3)</sup> 5 fibres/graticule area.

<sup>4)</sup> Sampling periods shorter than 10 min are not recommended.

### 3.9 Sampling Strategy and Records

Examples of strategy are given in Annex B. All data necessary for the determination of the fibre concentration shall be recorded, as well as sampling details. For an example of a sampling record (see Annex C).

## 4 EVALUATION

### 4.1 Sample Preparation

#### 4.1.1 Cleaning Slides and Equipment

Clean conditions shall be maintained at all times.

A dirty preparation area may result in sample contamination and erroneous results.

Clean slides with lens tissue or industrial paper tissue and lay them on a clean surface, for example, lens tissue sheet. It is good practice to clean each cover slip

with lens-tissue immediately before use, to ensure that the surfaces are free from contamination.

NOTE — Some types of lens-tissue can produce small fibres which may contaminate the preparation.

Wipe the scalpel and forceps with lens-tissue and place them on a clean surface, for example, lens tissue sheet. When mounting a series of filters, the mounting tools shall be wiped clean before dealing with each sample.

#### 4.1.2 Cutting the Filter Sample

Mounting of the total filter is preferable.

If it is necessary to cut the filter, all cutting should be done with a scalpel using a rolling action. Do not use scissors. It is recommended that the smallest piece mounted be wedge-shaped and approximately one-quarter or one-third of the filter.

#### 4.1.3 Mounting the Sample

For mounting, use the acetone-triacetin method as described in Annex D, unless a modified refractive index has to be used.

NOTE — Acetone mounting shall be carried out only in a fume hood or fume cupboard. Under no circumstances should it be conducted in the vicinity of an open flame.

### 4.2 Optical Requirements

#### 4.2.1 Microscope Equipment

Because microscopes with identical specifications can give quite different performances, it is necessary that the performance of proposed and existing microscopes be assessed by means of a detection limit test slide (see Annex E). Provided this criterion is met, small departures from the recommended specifications in item (d) and (e) are permitted. It is also important that newcomers consult experienced workers before selecting microscopes for fibrous dust determination. The necessary specifications are as follows:

- a) *Light source*, Kohler or Kohler type illumination. It is preferable for the illuminator to be built-in, but an external lamp with a plain mirror may be satisfactory. A variable light intensity control is necessary for both methods of illumination.
- b) *Substage assembly* — Abbe or achromatic phase-contrast condenser incorporated into a substage unit is required. There shall be a means of centering each condenser annulus with respect to the phase plate in the corresponding objective, and also a means of focussing the condenser.
- c) *Stage* — A built-in mechanical specimen

stage fitted with slide clamps and  $x - y$  displacement is required.

- d) *Objectives* — A rotating nose-piece fitted with  $\times 10$  and  $\times 40$  parfocal phase-contrast achromatic objectives. The  $\times 40$  objective shall have a numerical aperture (NA) of 0.65, achromatic. It shall have a phase ring of absorption not less than 65 percent and not greater than 85 percent.
- e) *Binocular eyepieces*, chosen to give a total magnification of 400 to 600. At least one eyepiece shall permit the insertion of a graticule. The compensating and focussing type are recommended. The use of body magnification changers is not recommended.
- f) *Graticule (Walton-Beckett)*, the diameter of the graticule in the object plane, when using the  $\times 40$  phase objective and an appropriate eyepiece shall be  $100 \pm 2 \mu\text{m}$ . See Annex F for graticule specification, calibration, source of supply and ordering information.
- g) *Accessories*
  - 1) Centering telescope or Bertrand lens for checking that the phase rings in the condenser are centered with respect to those in the objective.
  - 2) Green filter to ensure the best phase contrast conditions because the optics are designed for this wavelength.
  - 3) Stage micrometer which shall be subdivided into maximum  $10 \mu\text{m}$  intervals.
  - 4) Microscope slides shall be of optically homogeneous glass. It shall be  $75 \pm 1 \text{ mm}$  in length,  $25 \pm 1 \text{ mm}$  in width and 0.8 or 1.0 or 1.15 mm thickness. The variation in thickness shall be as minimum as possible.
  - 5) Cover slips of thickness (normally 0.17 mm) suitable for the microscope objective. Incorrect cover slip thickness will detract from the quality of the final image.
  - 6) Hand operated counter or similar device or a sheet with a print of 100 squares denoting 100 fields and the number of fibres counted in each field is entered into the square.

#### 4.2.2 Microscope Adjustment Principles

Follow the manufacturer's instructions while observing the following guidelines:

- a) Image of the light source shall be in focus and centered on the condenser iris of the annular diaphragm for true Kohler illumination.

- b) Object for examination shall be in focus.
- c) Illuminator field iris shall be in focus, centered on the sample and opened only to the point where the field of view is illuminated.
- d) Phase rings (annular diaphragm and phase shifting elements) shall be concentric.
- e) Eyepiece graticule shall be in focus. For more detailed information (*see* Annex G). Microscope adjustments shall be a daily routine.
- f) Before each counting exercise the resolution power of microscope shall be checked by detection limit test slide.

#### 4.2.3 Eyepiece Graticule Calibration

Each combination of eyepiece, objective and graticule shall be calibrated with a stage micrometer. Should any of the three be changed, the combination shall be recalibrated. For some microscopes, calibrations will change for observers with different interpupillary distances (*see* Annex F for eyepiece graticule calibration procedures).

#### 4.2.4 Microscope/Observer Performance Assessment

It is necessary that laboratories following this method should maintain contact with those having experience with it. As mentioned in 4.2.1 a detection limit test slide is available which will assist in the regular assessment of microscope and observer performance. A practical detection limit corresponding to block 5 on the HSE/NPL Test slide Mark II, shall be achieved (*see* Annex E for method of use and supplier). Exchange of microscope slides with experienced laboratories for comparison will help to ensure that valid results are being generated.

NOTE — It is desirable to determine intra-observer variation to assess reliability of an observer and in case, a laboratory employs more than one observer, inter-observer variation must be determined to standardize systematic variation in results.

### 4.3 Counting and Sizing Fibres

#### 4.3.1 Low Power Scanning

Scan the entire filter area with a total magnification of  $\times 100$  to  $\times 150$  (that is  $\times 10$  objective).

The margin normally covered by the filter holder gasket shall be free of dust and fibres. All viewing fields should have similar appearances with respect to total dust loading. If the observed fields show marked differences in loading or gross aggregation of fibres or dust, the filter shall be rejected. The filter shall also be rejected, in case the background dust is high and interferes with counting and sizing of

fibres or the filter shows any folds, kinks, or gross distortion due to improper mounting.

#### 4.3.2 Graticule Field Selection

After a satisfactory low power scan, change the microscope objective to  $\times 40$  phase and focus on the dust plane.

Ensure that the phase rings remain concentric. Although most of the fibres and dust will be found on the upper surface of the filter, it will be necessary to focus below (for example, up to  $10\ \mu\text{m}$ ) and slightly above the surface.

When counting and sizing, constant use of the fine focus is necessary because of the small depth of field of a  $\times 40$  objective (that is  $2$  to  $3\ \mu\text{m}$ ). Fields for counting shall be chosen at random throughout the entire area of the filter or filter segments. If the grid of a filter obstructs the view, move the stage to another field. Do not count fields that lie within  $3\ \text{mm}$  of the filter edge or within  $2\ \text{mm}$  of the cutting line, if any.

#### 4.3.3 Laboratory Working Conditions

The working practices and the working environment in a laboratory may influence systematically the level of reliability of the actual counting. This shall be controlled by a quality assurance scheme.

Some differences may appear when inter-laboratory comparisons are made which are due merely to different laboratory lighting conditions, different seating and computing arrangements, etc. Different ways of recording data may also cause some disagreement between the counters, due to the rate of visual fatigue.

The detailed writing of data involves the re-focussing of the eyes after viewing each field, whereas continuous registering with electrical or mechanical counters involves only a single period of continuous concentration.

#### 4.3.4 Counting Criteria

- a) *Choice of field* — Graticule areas for counting shall be chosen at random so that they are representative of the whole exposed area of the filter and do not overlap.

One method is to traverse the filter on randomly chosen chords taking fields at random.

- b) *Rejection of fields* — Graticule areas which include grid lines shall be rejected. If more than one-eighth of a graticule area is covered by an agglomerate of fibres and/or particles, the graticule area shall be rejected and another selected. Such occurrence shall be recorded.

- c) *Number of fibres and/or fields to be evaluated* — At least 100 fibres shall be counted with a minimum of 20 graticule areas evaluated. It is not necessary to evaluate more than 100 graticule areas.
- d) A countable fibre is defined as any object having a maximum diameter less than 3 µm, an overall length greater than 5 µm and a length : diameter ratio greater than 3:1, and which does not appear to touch any particle with a maximum diameter greater than 3 µm. Suitable pictures meeting the criteria (d) to (g) are given in equation 1.
- e) A countable fibre with both ends within the graticule area shall count as one; a countable fibre with only one end within the area shall count as half. An agglomerate of fibres which at one or more points on its length appears to be undivided but which at other points appears to divide into separate strands is known as a split fibre. Any other agglomerate in which fibres touch or cross one another is known as a bundle.
- f) A split fibre is evaluated as a single countable fibre, if it meets the definition in (d), the diameter being measured across the largest undivided part and not the split part.
- g) Fibres in a bundle area are evaluated individually, if they can be distinguished sufficiently to determine that they meet the definition in (d). If no individual fibres meeting this definition can be distinguished the bundle shall be evaluated as a countable fibre if it as a whole meets the definition.

#### 4.4 Calculation of Fibre Concentration

##### 4.4.1 Single Values

The fibre concentration  $c$ , in fibres/cm<sup>3</sup>, for each single sample duration is determined according to the following formula:

$$c = \frac{A}{a} \times \frac{N}{n} \times \frac{1}{r} \times \frac{1}{t}$$

where

- $A$  = effective filter area, in mm<sup>2</sup> (see Annex H);
- $a$  = graticule counting area, in mm<sup>2</sup> (see Annex F);
- $N$  = total number of fibres counted;
- $n$  = number of graticule areas observed;
- $r$  = flowrate of air through filter, in cm<sup>3</sup>/min; and
- $t$  = single sample duration, in min.

An example of a counting record is given in Annex J.

##### 4.4.2 Time-Weighed Average Values

When several samples of different sampling durations are taken, calculate the time-weighted average concentration  $C_{TW}$  in fibres per cubic centimetre, from the single values as follows:

$$C_{TW} = \frac{\sum c_i \times t_i}{\sum t_i} = \frac{c_1 \times t_1 + c_2 \times t_2 + \dots + c_n \times t_n}{t_1 + t_2 + \dots + t_n}$$

where

- $c_i$  = single value of concentration, in fibres/cm<sup>3</sup>;
- $t_i$  = single sample duration, in min; and
- $n$  = total number of samples.

If the single sample durations,  $t_i$ , referred to above are of equal duration, equation given in 4.4.2 can be simplified as follows:

$$C_{TW} = \frac{\sum c_i}{\sum t_i} = \frac{c_1 + c_2 + \dots + c_n}{n}$$

##### 4.4.3 Equivalent 8 h Exposure Value

If the shift of the worker exposed to airborne asbestos dust is more than or less than 8 h, the average concentration during the full shift shall be multiplied by a factor  $f$  to yield the equivalent 8 h exposure concentration  $C_{eq}$  as follows:

$$f = \frac{\text{Full shift time, in hours}}{8 \text{ h}} ; \text{ and}$$

$$C_{eq} = f \cdot C_{TW} (\text{full shift}).$$

##### 4.4.3.1 Calculation of $C_{eq}$ for various sampling schemes

4.4.3.1.1 For sampling scheme types A, B, C and D (see Annex B) calculate  $C_{eq}$  as follows:

- Calculate single value concentration(s) for the sample(s) using equation given in 4.4.1;
- Calculate time-weighted ( $C_{TW}$ ) concentration using the above single value(s) and respective single sample duration(s) using equations given in 4.4.2, as applicable; and
- Calculate  $C_{eq}$  by using the procedure given in 4.4.3.

4.4.3.1.2 For sampling scheme Type E (see Annex B), when 5 or more short term samples are taken at random throughout a full shift, the time-weighted average concentration shall be estimated as follows:

- Calculate the natural logarithm of each concentration as:

$$Y_i = \ln c_i, \text{ that is } y_1 = \ln c_1; y_2 = \ln c_2 \text{ and so on.}$$

If any concentration is  $< 0.1$  fibre/cm<sup>3</sup> replace it with 0.1 fibre/cm<sup>3</sup> for the above calculation.

- b) Calculate the arithmetic average of the logarithmic concentrations as follows:

$$\bar{y} = \frac{\sum y_i}{n} = \frac{y_1 + y_2 + \dots + y_n}{n}$$

- c) Calculate the empirical logarithmic standard deviation  $S_e$  of the logarithmic concentration(s) as follows:

$$S_e = \sqrt{\frac{1}{n-1} \left\{ \frac{\sum y_i^2 - 1 \sum (y_i)^2}{n} \right\}}, \text{ or}$$

$$S_e = \sqrt{\frac{1}{n-1} \left\{ y_1^2 + y_2^2 + \dots + y_n^2 - n(\bar{y})^2 \right\}}.$$

- d) The estimate of the average airborne concentration is calculated as follows:

$$C_{TW} = \text{Exp} \left( \bar{y} + \frac{S_e^2}{2} \right)$$

NOTE — The above calculations are used because random intra-day variations are best described by a logarithmic normal distribution.

- e) Using the value  $C_{TW}$  from above, calculate  $C_{eq}$  as given in 4.4.3.

**4.4.3.1.3** For sampling scheme Type F (see Annex B) calculate  $C_{eq}$  as follows:

- Calculate single value concentrations for the samples using equation given in 4.4.1;
- Calculate the times  $T_i$  for each individual working phase ensuring that the sum of these phase times equals a full shift;
- Calculate the time-weighted-average concentration using the phase times  $T_i$  instead of the single sample duration  $t_i$  in equation given in 4.4.2; and
- Calculate  $C_{eq}$  as given in 4.4.3.

NOTE — The above calculations for the different sampling schemes do not imply identical reliability in the estimation of the equivalent 8-hour exposure value  $C_{eq}$ .

## 5 SOURCES OF ERRORS

### 5.1 General

Errors introduced into the estimation of airborne fibres comprise sampling and analytical errors, each of which has a systematic and random component. The application of standard procedures and a reproducible routine (see Annex K) is the only way of controlling most of the many sources of error inherent in the membrane filter method. The following list describes some common sources of error.

## 5.2 Systematic Errors

### 5.2.1 Sampling

- Flowrate,
- Sampling time,
- Non-representative or biased sampling, and
- Contamination, deliberate or accidental.

### 5.2.2 Analytical

- Effective filter area,
- Counting area,
- Counting criteria,
- Filter mounting,
- Counting operator bias,
- Microscope, and
- Contamination.

## 5.3 Random Errors

### 5.3.1 Sampling

- Flowrate variability, and
- Random fluctuations of the airborne dust cloud.

### 5.3.2 Analytical

- Counting operator variability.
- Fibre distribution on the filter. Non-random deposition of dust on the filter leads to gross errors, the magnitude of which cannot be estimated. Twenty or more fields shall be counted to ensure that minor divergence from randomness does not bias the result.
- Poisson distribution.* As only small samples of the fibres deposited on the filter are counted, errors arise in the estimation of the total number of fibres on the entire filter face. Theoretically, the Poisson distribution defines the variation in fibre counts resulting from viewing randomly selected counting fields on the filter. If a minimum of 100 fibres is counted, and if a Poisson distribution were appropriate to the counting results, the relative standard deviation of the fibre counts would be  $\pm 10$  percent. It has been shown experimentally that the actual distribution of fibre counts can depart from that of Poisson, in which case the standard deviation may be greater.

## 5.4 Overall Accuracy

Because of the nature of the membrane filter method, it is not possible to know the true airborne fibre concentration of a given dust cloud. For this reason

it is not possible to assess the likely accuracy of the method. Even the precision or repeatability of the method is difficult to quantify because of systematic inter- and intra-laboratory errors which tend to arise. By randomly selecting observers and laboratories, these systematic errors take on a random nature so that it may be possible to provide estimates of empirical precision (that is, the closest possible approach to a statement of accuracy for a method with no known true values).

Much work has been done in an attempt to arrive at these estimates, and until now only partial conclusions have been reached. One of these conclusions is that the theoretical Poisson distribution (*see* 5.3.2) contributes a  $\pm 95$  percent confidence interval of  $\pm 20$  percent for a total of 100 fibres counted, or up to about  $\pm 35$  percent for only 40 fibres counted in 100 graticule areas.

Other sources of random and systematic errors add significantly to the uncertainty in estimating the airborne fibre concentration.

### 5.5 Limitations of the Membrane Filter Method and Presentation of Results

With the parameters specified in this method, that is, 1 l/min flowrate and a minimum filter loading of 15 fibres/100 graticule areas, the theoretical low detection limit for an eight-hour sample is 0.02 fibres/cm<sup>3</sup>, however, the practical limit is much higher. It is generally accepted that blank unused filters may frequently give a reading of several countable fibres per 100 graticule areas. These fibres may be unidentified contaminants on the filter or artifacts from the cleaning process which have the appearance of fibres. It is recognized that neither counting more fields nor increasing sampling duration overcomes

the problems of background dust, when asbestos is a minimum constituent in the overall dust cloud.

There is at present insufficient information available to determine at what level the reliability of the method becomes so poor that results have little meaning. It is clear that this will not be a single value, but will be a range depending upon at least the relative and absolute fibre concentration. There appears to be general agreement amongst those experienced in the field that these limits lie somewhere in the range of 0.1 to 0.5 fibres/cm<sup>3</sup> depending on a variety of conditions. In view of this situation and the inherent variability of the method, all calculated values of less than 0.1 fibre/cm<sup>3</sup> shall be reported only as less than 0.1 fibres/cm<sup>3</sup>. All higher values shall be rounded off to the first decimal place, and to two significant figures.

## 6 TEST REPORT

The test report should include the following information:

- a) Reference to this standard;
- b) Sample identification number;
- c) Start and end of the sampling period;
- d) Flowrate during the sampling period;
- e) Type of sample: personal or static sample;
- f) Description of the location where the sample was taken;
- g) Results;
- h) Any deviations from the sampling and the analytical procedure; and
- j) Any other information relevant to the method.

An example of a sampling record is given in Annex C.

## ANNEX A

### ( Clause 3.5 )

#### FLOWRATE CALIBRATION AND CORRECTIONS

##### A-1 GENERAL

Internal and external flow-meters should be calibrated with a primary calibration device. A method of calibration of the commonly used variable area flow-meter (that is, rotameter) using the soap film flow-meter is described in this annex.

##### A-2 PROCEDURE

Choose an accurate burette (or similar) of capacity 300 to 1 000 cc. Attach a tube to the bottom of the burette, and then clamp it into a stand in an inverted vertical position.

Set up the sampling pump equipped with connecting tube, filter holder and filter as used in the field (see Fig. 2).

Connect the soap film flow-meter. Ensure that the system is leak-proof. It is advisable to rinse the burette thoroughly in water immediately before the test. This removes accumulated detergent and also assists in wetting the inside of the burette.

Switch on the pump and adjust the flowrate according to the internal flow-meter (if fitted).

Partly fill a beaker or petri dish with water plus the minimum amount of detergent necessary to permit bubbles to be formed.

By momentarily placing the beaker against the bottom of the soap film flow-meter, create a bubble that will travel the entire length of the burette without bursting.

With a stopwatch, measure accurately the time that the bubble requires to traverse the tube between its extreme graduated ends.

Repeat the last two steps and at least twice, until good repeatability of the times is achieved.

Average the times, and calculate the true volumetric flowrate,  $q_c$ , in  $\text{cm}^3/\text{min}$ , under calibration conditions appropriate to the sampling conditions, as follows:

$$q_c = \frac{V}{t}$$

where

$V$  = volume of the burette, in  $\text{cm}^3$ ; and

$t$  = average time in min, required for the bubble to transverse the tube, in min.

Repeat the first nine steps of the procedure and the calculation until the desired flowrate has been reached within 5 percent.

If the external or internal variable area flow-meter is used under temperature conditions which differ from those used during calibration, it is generally not possible to calculate the different flowrate that will inevitably result.

As all air sampling measurements are concerned only with volumetric flowrate ( that is, flowrate measured and expressed at the prevailing temperature and pressure ) and not mass flowrate ( that is, flowrate corrected to standard temperature and pressure conditions ), re-calibration of the pump flowrate is essential, if it is operated under conditions substantially different from those of calibration. Substantial implies a difference in altitude or temperature of more than 500 m or  $15^\circ\text{C}$  respectively compared to the calibration conditions.

NOTE — Theoretically, the water vapour content of the air in the soap film flow-meter should be taken into consideration when determining the true flowrate. However, for practical purposes, acceptable accuracy is maintained without this correction.

##### Example

During the calibration of a pump with an internal flow-meter, a soap film flow-meter of 1 000 cc volume gave an average of 63.4 s for the bubble to traverse its length. Calculate the flowrate under these conditions. Using equation given in this annex :

$$q_c = \frac{V}{t} = \frac{1000}{63.4/60} = 946 \text{ cc/min}$$

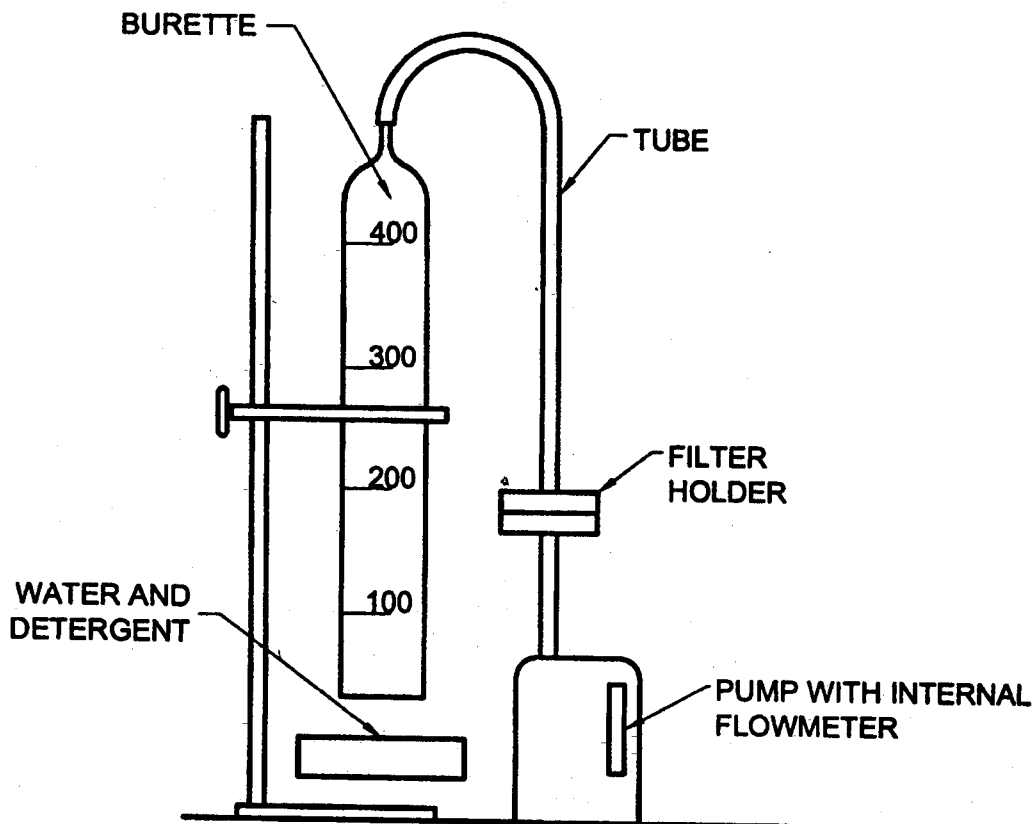


FIG. 2 FLOWRATE CALIBRATION APPARATUS

## ANNEX B

( *Clauses 3.9, 4.4.3.1.1, 4.4.3.1.2 and 4.4.3.1.3* )

### EXAMPLES OF SAMPLING STRATEGY

#### B-1 TERMINOLOGY

##### B-1.1 Occupational Sampling

Sampling conducted so that the results are representative of the worker's exposure to fibres under typical working conditions for a full shift. Sampling procedures should not interfere with the activities of the worker.

##### B-1.2 Worker's Breathing Zone

Consists of a hemisphere of 300 mm radius extending in front of the face, and measured from the midpoint of a line bisecting the ears.

In order to estimate the worker's exposure, samples should be taken in the worker's breathing zone.

##### B-1.3 Personal Sample

Sample taken within the worker's breathing zone.

Usually the filter is fastened to the lapel of the worker's jacket with the cowl pointing downwards, in case a cowl is used. The worker carries the pump on a belt or in a pocket.

##### B-1.4 Static Sample

Sample taken at fixed locations with cowl pointing downwards. They are not recommended for the measurement of personal occupational exposure.

Point sources create considerable concentration gradients, thus causing the results of static sampling to vary considerably over short distances. However, static sampling can be useful if the dust is proven to be uniformly distributed over large areas.

##### B-1.5 Single Sample Duration

The actual time during which a single sample is collected.

### B-1.6 Total Sample Duration

The sum of single sample durations taken during one shift (see B-2.3) on one person.

### B-1.7 Short-Term Sample

Sample with a single sample duration of less than 1 h (see 3.8).

The short-term sample was defined because it is necessary to refer to this special case. Unless specified as short-term, sampling is assumed to be of at least 1 h.

## B-2 STRATEGY

### B-2.1 General Principles

Occupational exposure measurements are carried out to meet one or both of two major objectives:

- To assess exposure relative to an occupational hygiene standard and to enable better control measures to be implemented; and
- To provide estimates of exposure for epidemiological investigations of morbidity and mortality.

It is well-known that the concentrations vary widely both within a single day and from day-to-day. Most regulations and hygiene standards require a reliable estimate of exposure on a particular day. It is more useful for epidemiology to spread the sampling effort over a number of days, that is, less will be known about a single day, but more about the average exposure over a working lifetime. Since sampling often serves both purposes, the sampling schemes presented in this method emphasize the single day estimate. It should also be recognized that variations in individual working practices result in a distribution of exposure values within any working group. Consequently, data from one person cannot be assumed to be representative of the total working group. Any transfer of data should, therefore, be validated by appropriate relative measurements.

### B-2.2 Sampling Scheme

There are a number of possible sampling schemes, some of which are listed for guidance in Tables 2 and 3. As the schemes vary in the degree of usefulness and precision in estimating exposure, Tables 2 and 3 should be interpreted in association with the qualifying conditions and cautions presented in B-2.3 and B-2.4.

**B-2.2.1** In planning a sampling scheme, it is important to determine;

- Estimation period during which the exposure is estimated;

**Table 2 Long-Term Sampling Scheme**

( Clause B-2.2 )

Sampling Scheme	Number of Samples per Shift	Total Sampling Duration
(1)	(2)	(3)
Full-shift consecutive sample(s)		
Type A	2 or more	Approximately full-shift
Type B	1	Approximately full-shift
Partial-shift consecutive sample(s)		
Type C	2 or more	2 h or greater
Type D	1	1 h or greater

**Table 3 Short-Term Sampling Scheme**

( Clause B-2.2 )

Sampling Scheme	Number of Samples per Shift	Total Sampling Duration
(1)	(2)	(3)
Random Samples		
Type E	5 or more taken randomly throughout the working day	1 h or greater
Systematic Samples		
Type F	1 or more plus continuous relative measurement, or 2 or more taken during each separate phase of a cyclical operation	1 h or greater

- Total sample duration; and
- Number of samples.

To assess the worker's full shift exposure, every effort shall be made to ensure that the samples relate to a whole working day. Care should be taken to ensure that the sampling period is not biased by abnormal conditions.

Short-term samples should be taken at random ( statistically ) throughout the whole working day. If samples cannot be selected from the entire working day, the measurement results are valid only for the duration of the period from which the measurements were selected. However, relative measurements and reliable professional judgement can sometimes be used to make inferences about concentrations during other portions of the day. Reliable knowledge of the operation is essential when making such extrapolations.

### **B-2.3 Total Sampling Duration and Number of Samples**

Sample duration is influenced primarily by the reason for sampling, the level of fibre concentration to be measured, the concentration of non-fibrous dust and the requirements of the analytical method. This may result in more than one single sample being required. The total sample duration should never be less than 1 h.

3.6 and 3.8 give the details of acceptable minimum and maximum loadings of fibres on the filter, which dictate the range of possible sampling times for different air-borne fibre concentrations. Samples of short duration may be necessary if high background levels of particulate matter or fibres which would prevent accurate analysis are present.

### **B-2.4 Reliability of Sampling Schemes**

The main strength and limitations of the various sampling schemes, Types A to F listed in Tables 3 and 4 are as follows.

#### **B-2.4.1 Type A Sampling Scheme**

Two or more samples covering the full working shift.

This permits the most reliable estimate of exposure to be made. When several samples are taken, the average of the errors is usually less than the single (percentage) error in a single full-shift sample. Occasional gross errors (such as, miscalculations, contamination, incorrect sample timing, etc) are more likely to be detected by Type A than by Type B.

NOTE — Systematic errors, for example, flowrate inaccuracy, should still be taken into account in the normal manner.

#### **B-2.4.2 Type B Sampling Scheme**

One full-shift sample.

This is not as reliable as Type A, because gross errors can escape detection unless evidence from previous sampling is available on which to base a judgement.

#### **B-2.4.3 Type C Sampling Scheme**

Two or more samples covering part of the full-shift, that is, 2 h or greater but less than the full-shift.

This can be satisfactory if the partial shift is representative of the full shift.

#### **B-2.4.4 Type D Sampling Scheme**

One sample 2 h or greater but less than full-shift.

This is similar to Type C except that gross errors may escape detection.

#### **B-2.4.5 Type E Sampling Scheme**

Five or more short-term samples, taken randomly throughout the full-shift.

This may give an acceptable indication of exposure but is generally more wasteful of resources and is the least precise of the above schemes. Note that an even poorer estimate results when the average dust concentration increases or decreases markedly throughout the day. This scheme should be applied with caution.

#### **B-2.4.6 Type F Sampling Scheme**

Several short-term systematic samples taken during each separate phase of an operation.

This can be used by experienced industrial hygienists to characterize a workplace. This approach should not be used indiscriminately, particularly by persons not completely familiar with the process. Nor should it be used to estimate time-weighted-average exposure, unless the results are verified by continuous relative measurements or other methods (see B-2.2).

### **B-2.5 Recommended Frequency of Sampling**

It is difficult to prescribe general rules for frequency of sampling as it depends on the purpose of sampling. For routine work environment monitoring, the guiding factors are legislative requirements and expected dust concentration in work environment. The following guidelines may be used for this purpose:

- a) Once in a month where asbestos dust concentration is likely to exceed prescribed exposure limit occasionally;
- b) Once in three months where asbestos dust concentration is likely to be between exposure limit and action level;
- c) Once in every 6 to 12 months where asbestos dust concentration is below action level, and
- d) Once in every 12 months at all workspots where there is asbestos exposure irrespective of dust concentration.

**ANNEX C****( Clauses 3.9, 6 and K-1.4.6 )****DUST SAMPLING RECORD**

**C-0** All data necessary for the determination of the fibre concentration should be recorded on a sampling record. Furthermore, all available data which may be of value for epidemiological studies should be included.

**C-1 SAMPLING DETAILS**

- a) Instrument type and number;
- b) Flowrate (initial, intermediate and final);
- c) Duration;
- d) Sampling scheme used;
- e) Date, hour; and
- f) Sampled by.

**C-2 SAMPLING PLACE DETAILS**

- a) Designation,
- b) Harmful substances,
- c) Types of asbestos,
- d) Brief description of working process,
- e) Variable parameters which can exercise an influence on dust formation.
- f) Work practices:
  - 1) Working conditions:

- i) normal, and
- ii) abnormal.
- 2) Material:
  - i) type,
  - ii) size, and
  - iii) conditions, etc.
- 3) Airflow:
  - i) worker in dust airflow, yes/no; and
  - ii) obvious influence on adjoining working places.
- 4) Methods of dust control:
  - i) exhaust ventilation,
  - ii) other methods, and
  - iii) visual impression.
- 5) Number of employees for which the measuring value is representative:
  - i) personal protection yes/no, type,
  - ii) hours per shift; and
  - iii) days per week.

An example of a dust sampling record is as follows:

Dust Sampling Record ( Example only )

Place of measurement

Date

Measuring point/Name

.....

Code No.

Text in clear

Dimension of workplace

< 50m<sup>3</sup>

50 m<sup>3</sup> to 500 m<sup>3</sup>

500 m<sup>3</sup> to 5 000 m<sup>3</sup>

> 5 000 m<sup>3</sup>

Exhaust ventilation

yes

no

Situation representative

yes

no

Dust concentration

above average

below average

Visual impression

good

quite good

bad

Number of employees working at this working place :

.....

Respirators are worn

yes

no

sometimes

Type

.....

Draught during measurement

no

yes

.....

Measured in the dust-laden air flow

yes

no

Adjoining working places are influenced

no

yes

Measuring point No.

.....

Measurement was done

personal

static

Sampling device

Atmospheric pressure

mbar

Air flowrate

time started

time ended

Sampling scheme used

Sample No.	Sampling time (min)	Total flow	Working phase	fibres/cm <sup>3</sup>
	.....	.....	.....	
	.....	.....	.....	
	.....	.....	.....	
	.....	.....	.....	
	.....	.....	.....	
	.....	.....	.....	
	.....	.....	.....	
	.....	.....	.....	

Average value

Harmful substances

Chrysolite

crocidolite

Amosite

.....

(other)

Other fibres

Glassfibre

Mineral wool

.....

(other)

## ANNEX D

### ( Clause 4.1.3 )

#### ACETONE-TRIACETIN MOUNTING PROCEDURE

##### D-1 GENERAL

The following is a description of a procedure for mounting membrane filters. The device used is largely composed of parts available in chemistry laboratories. Other methods using commercially available apparatus like acetone vaporizer, a light weight portable apparatus, may be used when they produce samples of the same (or better) quality. The handling of acetone requires great care in order to avoid accidents. Even safer devices are now being developed. They may be used, provided that the same slide quality (no washing off of fibres, smooth surface, clear background) can be obtained with them.

**WARNING** — Acetone mounting should be carried out only in a fume hood or fume cupboard. On no occasion should it be conducted in the vicinity of an open flame. Only a small quantity of acetone is necessary. Heating with the water bath is preferable; use of boiling chips in the acetone is recommended.

As illustrated in Fig. 3, it is advisable to use a simple condensing column to ensure that a bare minimum of acetone vapour escapes. The free opening in the tap shall have a diameter of at least 8 mm, otherwise the acetone vapour cannot escape in sufficient quantity when using an open condensing column. When the apparatus is not in use, the acetone vapour outlet should be closed.

##### D-2 PROCEDURE

- a) Heat the acetone to boiling and wait until a moderate quantity of acetone vapour emerges from the outlet.
- b) Place the filter, dust side up, on a clean microscope slide at room temperature. Electrostatic forces usually keep the filter on the slide.
- c) Ensuring that no liquid acetone drips onto the filter (by wiping the outlet periodically with a tissue), hold the slide with clean forceps directly in the acetone vapour, stream, approximately 15 to 25 mm from the outlet for 3 to 5 s. At the same time, move the filter slowly across the outlet to ensure even coverage until the filter is transparent. Too little vapour will fail to render the filter transparent, while too much vapour (especially drops of liquid acetone) will destroy the filter by dissolving it or shrinking it beyond use. The slide shall not be pre-warmed, as it is necessary for acetone vapour to condense on the slide for correct clearing.
- d) Using a hypodermic syringe with a 22-gauge needle or a disposable micropipette, place 1 to 3 drops of glycerol triacetate (triacetin) on the acetone-cleared filter. To avoid the development of a skin over the triacetin, immediately lower a clean cover slip onto the triacetin at an angle (*see* Fig. 4). The cover slip should not be pressed onto the membrane.
- e) Too much triacetin (as indicated by excess liquid emerging from the edges of the coverslip) can cause the outside edge of the filter eventually to disintegrate to some degree. Insufficient triacetin will result in uneven clearing of the granularity left from the acetone vapour clearing. Furthermore, the refractive index of the mounted sample will not be suitable for optimum visibility for some fine fibres.
- f) Heating the cleared filter to approximately 50°C for 15 min accelerates the clearing process and enables the analysis to proceed almost immediately thereafter. Otherwise, it is necessary to delay counting for about 24 h until the entire filter has dissolved under the action of the triacetin. The finished product will be permanent.
- g) The edge of the coverslip may be sealed with lacquer varnish (for example, nail polish), if the slide is to be kept indefinitely.

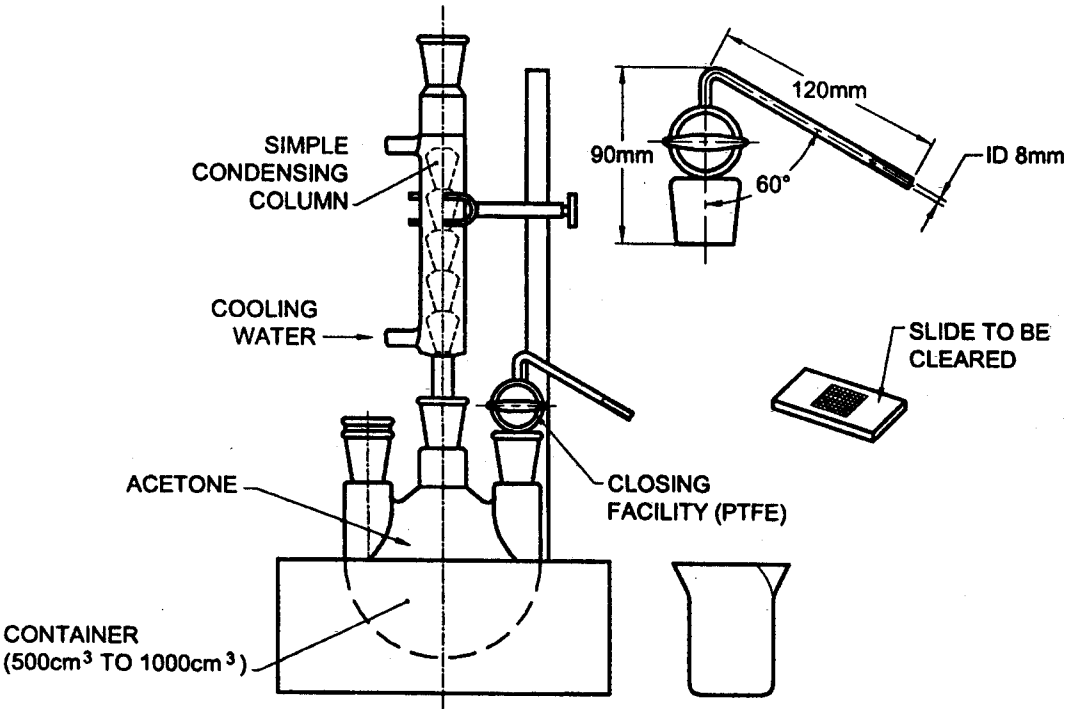


FIG. 3 CONDENSING COLUMN

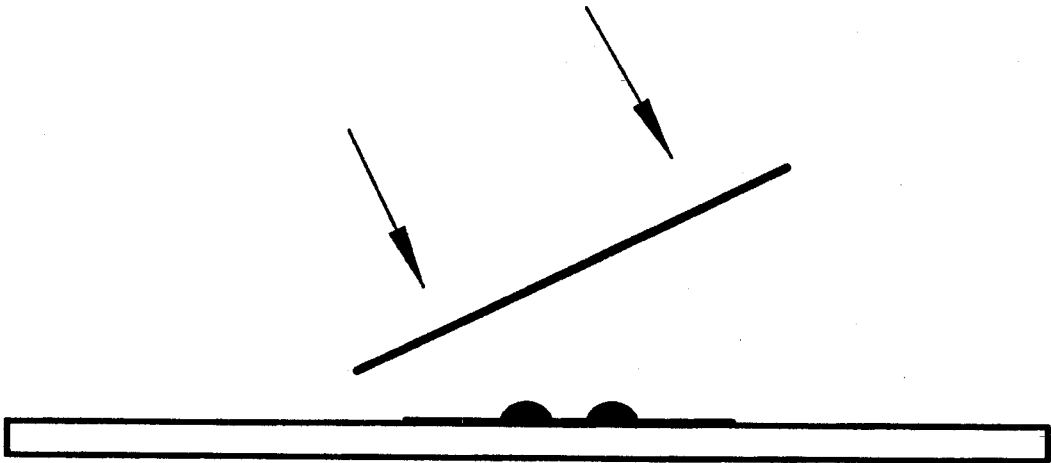


FIG. 4 PLACING OF THE COVER SLIP

## ANNEX E

( *Clauses 4.2.1, 4.2.4 and K-1.4.3* )**HSE/NPL TEST SLIDE (MARK II)<sup>1)</sup> FOR THE DETERMINATION OF THE DETECTION LIMIT  
WHEN USING PHASE CONTRAST MICROSCOPY****E-1 DESCRIPTION**

The standard HSE/NPL test slides consist of identical epoxy replicas (with a refractive index of 1.58 ) of a master slide produced and certified by the National Physical Laboratory (U.K.). The epoxy replicas are mounted on a glass slide of dimensions 75 mm × 25 mm × 1.2 mm or 75 mm × 25 mm × 0.8 mm, and covered by a coverslip 0.17 mm thick with a layer of another resin with a refractive index of 1.49 in-between. The test objects consist of a series of seven blocks of ridges of length 8.5 mm filled with a resin of refractive index 1.49 in a medium of refractive index 1.58. The ridges have a V-shaped profile and have a height to width ratio of about 0.1. The blocks are separated by gaps 20 µm wide. A set of four deep marker ridges is placed on either side of the array and a further two sets of two marker ridges, spaced at an interval of 120 µm, intersect the array at right angles. The zone of the test objects to be used is delineated by the rectangle bounded by these marker ridges. This zone can easily be located as the field of view in which it is found, and is engraved on the coverslip. This is illustrated in Fig. 5. The widths of the ridges within each block and the calculated phase change (in degrees), associated with the maximum path difference in light rays passing through the test objects, are given in Table 4.

**E-2 METHOD OF USE**

Set up the microscope for phase contrast microscopy

**Table 4 Widths of Test Objects and Calculated Maximum Phase Change Induced in Light Rays Passing Through Test Objects of HSE/NPL Test Slide**

( *Clause E-1* )

Block Number	Ridge Width µm	Maximum Calculated Phase Change (in Degrees) for Light Rays <sup>1)</sup> Passing Through Test Objects
(1)	(2)	(3)
1	1.08	6.6
2	0.77	4.7
3	0.64	3.9
4	0.53	3.2
5	0.44	2.7
6	0.36	2.2
7	0.25	1.5

<sup>1)</sup> Wavelength: 530 nm.

as recommended for the membrane filter method (see 4.2.1).

Locate block I (the coarsest set, see Table 2) of the test objects and move the slide to observe adjacent blocks. Determine the block of the finest ridges that can be distinguished. It is unlikely that all seven blocks of ridges will be detected using optical phase contrast techniques, even on the best research microscope. On the basis of present information, a satisfactory system will detect block 5.

<sup>1)</sup> HSE/NPL test slide (Mark II) is the trade-name of a product supplied by PTR OPTICS, Unit D9. Cross Green Approach, Cross Green Industrial Estate, Leeds, Yorkshire, LS OSG, England. This information is given for the convenience of users of this standard and does not constitute an endorsement by BIS of the product named. Equivalent products may be used, if they can be shown to lead to the same results.

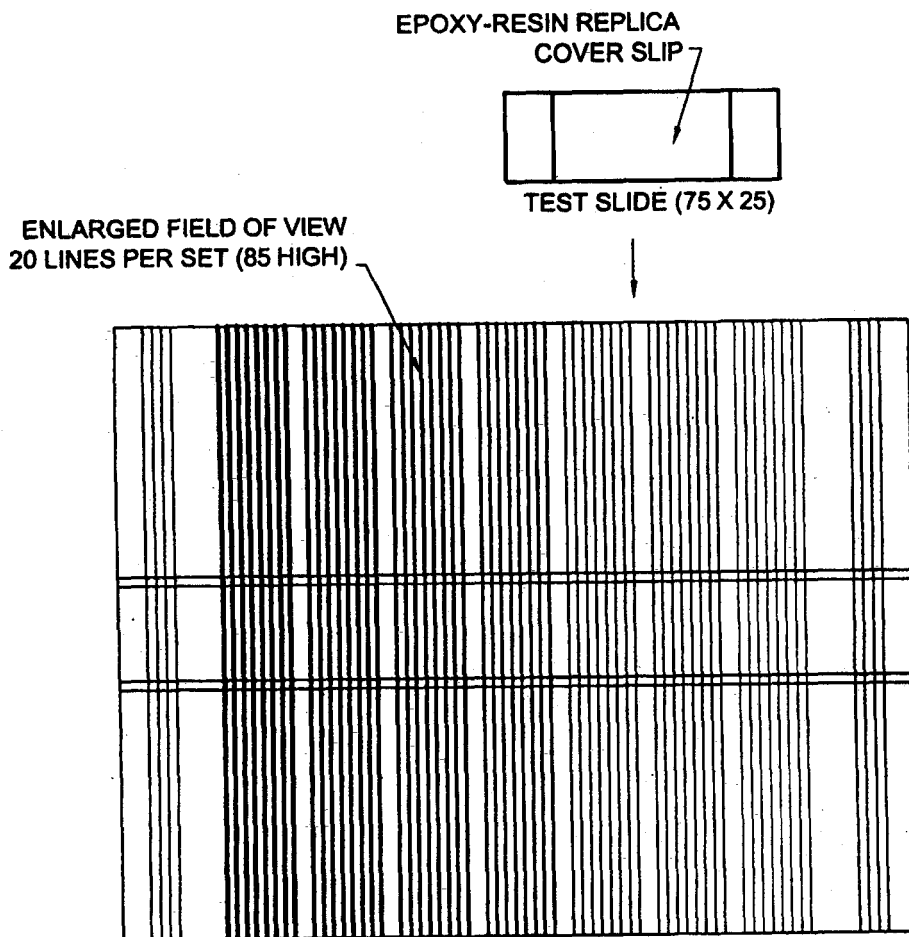


FIG. 5 HSE/NPL TEST SLIDE

## ANNEX F

( *Clauses 4.2.3 and 4.4.1* )

### EYEPiece GRATICULE

#### F-1 SPECIFICATIONS OF EYEPiece GRATICULE, ORDERING INFORMATIONS AND CALIBRATION

The graticule described in this method is the type G22 Walton/Beckett graticule<sup>1)</sup>.

For each graticule, the desired diameter,  $d$ , of the circle to appear as  $100 \pm 2 \mu\text{m}$  in the object plane  $D$  of the graticule, and the overall diameter of the glass

disc should both be specified in millimetres before ordering.

The following procedure is one of several methods for determining the diameter,  $d$  of the circular counting area:

- Insert any available graticule into the eyepiece and focus so that the graticule grid is sharply in focus.
- Set the appropriate inter-pupillary distance, and if applicable, reset the binocular head adjustment so that the tube length ( and thus the magnification ) remains constant.
- Ensure that the  $\times 40$  phase objective is in place, and that the magnification changer position ( if used ) is known and recorded.

<sup>1)</sup> Type G22 'Walton/Beckett' graticule ( Reference No. G22 ) is the trade-name of a product supplied by Graticules Limited, Sovereign Way, Botany Trading Estate, Tonbridge, Kent, TN 9 1RN, England. This information is given for the convenience of users of this Standard and does not constitute an endorsement by BIS of the product named. Equivalent products may be used, if they can be shown to lead to the same results.

- d) Place a stage micrometer on the microscope object stage and focus the microscope onto the graduated line.
- e) Measure the overall object length,  $l_o$ , of the graticule grid using the stage micrometer.
- f) Remove the graticule from the microscope and measure its actual overall grid length,  $l_a$ . This can be done by using a stage fitted with verniers.
- g) Calculate the diameter to be specified,  $d$ , using the following equation:

$$d = \frac{l_a}{l_o} \times D$$

### Example

**Step (e) produced an object length of a Porton graticule of 108  $\mu\text{m}$ .**

Step (f) produced an actual length of 4.50 mm.

Step (g) produced a diameter of  $(4.50/0.108) \times 0.1 = 4.17$  mm.

It is also necessary to measure the overall diameter of the glass disc.

**In this case the disc diameter was found to be 17 mm. Thus a Walton/Beckett graticule of disc diameter 17 mm and circle diameter 4.17 mm should be specified for the above example.**

## F-2 CALIBRATION OF EYEPIECE GRATICULES

- Obtain a stage micrometer, preferably with a scale having 2  $\mu\text{m}$  or 10  $\mu\text{m}$  divisions and place it on the object stage of the microscope.
- Make sure that the inter-pupillary distance of eyepieces is set correctly.
- Note the objective magnification and any

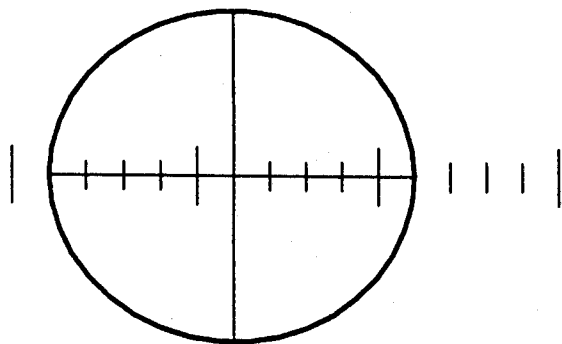


FIG. 6 SUPERIMPOSED EYEPiece GRATICULE AND STAGE MICROMETER

intermediate magnification used.

- d) Focus the microscope onto the graduated marks of the stage micrometer.
- e) Line up the eyepiece graticule with the graduated divisions on the micrometer, so that the number of whole micrometer divisions can be counted from one side of the eyepiece graticule graduations to the other.
- f) If less than a whole division remains, estimate this fraction to the nearest micrometer and add it to the number of whole divisions of the stage micrometer after converting to micrometers. This totalled result is the projected or object dimension of the eyepiece graticule.

### Example

- A stage micrometer with 10  $\mu\text{m}$  divisions was placed on the stage of a microscope.
- The diagram in Fig. 6 depicts the view of the superimposed eyepiece graticule and stage micrometer.

**Note that 10 whole divisions span across the graticule, that is,  $10\text{ }\mu\text{m} \times 10\text{ }\mu\text{m}$ .**

- c) The remainder of the 11th division is estimated as being one third of a whole division, that is, approximately  $3\text{ }\mu\text{m}$ .

Adding the values in (b) and (c) together gives 103  $\mu\text{m}$  which is the object dimension of the eyepiece graticule. Note that if the interpupillary distance, objective, intermediate magnification, or if in some microscopes the eyepiece is changed, then this usually changes the object dimension of the eyepiece graticule, thus necessitating recalibration.

**Fig. 7 illustrates a Walton-Beckett Graticule.**

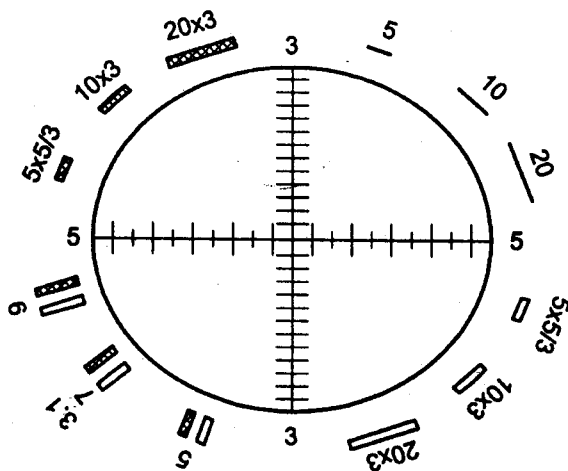


FIG. 7 WALTON-BECKETT GRATICULE

## ANNEX G

### ( Clause 4.2.2 )

#### MICROSCOPE ADJUSTMENT PROCEDURE

**G-1** Good quality phase-contrast microscope equipment should be used as detailed in 4.2.1. The equipment should be maintained in good condition and most manufacturers operate a routine maintenance service, which includes the stripping down and cleaning of all optical components and the replacement of worn traverse mechanisms. Such services should be used unless skilled maintenance services can be provided by counting-laboratory staff.

In general, the following setting-up procedure should be adopted to obtain Kohler illumination and good phase-contrast conditions. However, the details may vary according to the manufacturer's instructions and the type of equipment.

- a) Place the membrane filter specimen slide on the microscope stage.
- b) Open both the illuminator diaphragm ( often referred to as the field iris ) and the substage condenser diaphragm.

NOTE — At this stage the phase annuli should not be inserted. These are usually placed in a rotating drum fitted into the substage condenser unit.

- c) Raise the condenser to its upper limit, usually within 1 mm of the lower face of the specimen slide.
- d) Using a convenient level of illumination and the  $\times 10$  objective, focus the specimen,
- e) Close down the illuminator diaphragm and focus this in the field of view by lowering and raising the condenser. Centre the diaphragm and re-open it to fill the field of view.

- f) Observe the back focal plane of the objective, using either a Bertrand lens fitted to the body of the microscope or by removing the eyepiece and using an auxiliary telescope.
- g) Observe the image of the bulb ( removing the diffusing disc, if one is fitted ) and centre the bulb filament, focussing the bulb, if possible with the adjustment provided. The image of the bulb filament should fill the back focal plane of the objective. Reinsert the diffusing disc if appropriate.

NOTE — If the bulb cannot be focussed, adjust to give uniform bright illumination.

- h) Insert the correct phase annulus into the condenser system and centre this using the appropriate adjusting screws so that the phase plate in the objective and the image of the annulus coincide exactly. If necessary, adjust slightly the condenser focussing. Ensure that the bright annulus image does not extend beyond the phase ring.
- i) Revert to normal viewing and change to the  $\times 40$  objective with no phase annuli in the condenser system. Close down the field diaphragm and refocus this by appropriate adjustment of the condenser. Re-centre if necessary and re-open to fill the field of view.
- k) Repeat steps (f) and (h) after inserting the phase annulus appropriate to the  $\times 40$  objective.
- m) Revert to normal viewing.

## ANNEX H

### ( Clause 4.4.1 )

#### MEASUREMENT OF EFFECTIVE FILTER AREA

**H-1** Effective filter area may be determined by either of the two methods described in **H-1.1** and **H-1.2**.

##### **H-1.1 Method 1**

One convenient method of determining the effective filter area is as follows:

- a) Place a small quantity of dark coloured fine dust (for example, carbon, cement or road dust) into a 2 to 5 litre container with lid.
- b) Shake the container, remove the lid and draw air through a membrane filter and its holder until the airborne dust in the container forms an obvious deposit on the filter.
- c) Remove the filter from the holder and mount onto a microscope slide in the normal manner.
- d) Measure at least four different diameters of the resultant dust spot to within 0.2 mm. Amongst other methods, micro-projection measurement or the use of microscope object stage verniers have been found satisfactory.
- e) Provided that the measured diameters differ by not more than 1 mm, a simple arithmetic average is sufficient to provide a good estimate of the effective filter diameter.
- f) At least three individual filters shall be prepared and mean area calculated.
- g) Provided that the three filter diameters differ not more than 1 mm, an arithmetic average should be taken and the area calculated in the usual manner. This area is then the effective

filter area.

- h) If steps (e) or (g) produce differences greater than 1 mm, close attention should be paid to the sampling of the dust or to the filter clearing technique.
- j) It is necessary to repeat the measurement of the effective filter area if the type of filter or holder, or if any aspect relating to filter clearing is changed.
- k) It is advisable to repeat the entire measurement procedure every 12 months to ensure that the correct effective filter area is known.

##### **H-1.2 Method 2**

A flame which produces excessive smoke is used in this method to determine effective filter area. For this purpose a spirit lamp with diesel or mobile oil may be used. The following procedure shall be followed:

- a) Light the flame and wait till it produces heavy smoke. Keep filter holder directly in smoke at sufficient distance ( approximately 200 mm ) to avoid burning.
- b) Rotate the filter holder slowly and allow smoke particles to deposit in a uniform layer on the filter.
- c) Process the filter in the similar way as described in **H-1.1** ( c ) to ( k ).

**H-1.2.1** This method is simple, quick and gives better delineation of effective filter area.

ANNEX J  
( Clause 4.4.1 )

EXAMPLE OF A DUST COUNTING RECORD

DUST COUNTING RECORD (Example only)							
Counted by :	<table><tr><td colspan="2">Number of</td></tr><tr><td>fibres</td><td>fields</td></tr><tr><td></td><td></td></tr></table>	Number of		fibres	fields		
Number of							
fibres		fields					
Date :							
Microscope No. :							
Graticule type :							
0 – bundles	Area mm <sup>2</sup>						
× – background not okay							
$c = \frac{\sum n_f}{\sum n_{cf}} \times \frac{1}{V} \times F = \dots\dots\dots =$							
	<table><tr><td></td><td>Fibres/cm<sup>3</sup></td></tr></table>		Fibres/cm <sup>3</sup>				
	Fibres/cm <sup>3</sup>						

In special circumstances it is of value to record the number of fibres contained in individual fields of counting.

EXAMPLE

Sample No.


$c = \frac{\sum n_f}{\sum n_{cf}} \times \frac{1}{V} \times F = \dots\dots\dots =$	<table><tr><td></td><td>Fibres/cm<sup>3</sup></td></tr></table>		Fibres/cm <sup>3</sup>
	Fibres/cm <sup>3</sup>		

- $c$  is the concentration in fibres/cm<sup>2</sup>
- $N = \sum n_f$  is the total number of fibres counted
- $n = \sum n_{cf}$  is the number of fields counted
- $A$  is the effective filter area, in mm<sup>2</sup>
- $a$  is the area of the counting field, in mm<sup>2</sup>
- $V$  is the total flow, in cm<sup>3</sup>
- $F = \frac{A}{a}$  (constant factor)

## ANNEX K

## ( Clause 5.1 )

## ROUTINE FOR SAMPLING AND ANALYSIS

**K-1** It is important to follow a reproducible routine to minimize variation in results which may be introduced by day-to-day factors. Following routine may be used as a guide.

**K-1.1 Preparation**

**K-1.1.1** The samples shall be collected according to a plan evolved on the basis of preliminary studies.

**K-1.1.2** Ensure a day earlier that sampler is fully charged, in case the battery is weak it shall be left for charging overnight or the time specified by the manufacturer.

**K-1.1.3** Before proceeding for sampling, check that pump is in working order.

**K-1.1.4** Check that all connections are leak-proof.

**K-1.1.5** Mount filter in filter holder and check that filter is properly placed and does not show any distortion or cracks.

**K-1.1.6** Adjust flowrate to 1.0 l/min with the help of external flow meter or pre-calibrated internal flow meter.

**K-1.1.7** If external rotameter shows visible pulsation, connect an external damper.

**K-1.1.8** If more than one filter holder is to be used, label each filter holder.

**K-1.1.9** Cover all filter holders with plastic caps to prevent contamination with dust.

**K-1.1.10** It is always a good practice to place all equipment in sampling kit and take it to the plant.

**K-1.2 Sampling**

**K-1.2.1** Survey the area to be sampled and surroundings for any abnormal working conditions, such as dust emission from nearby machines, operation of dust collectors, sweeping in progress, movements of in-plant vehicles, etc.

**K-1.2.2** Note the presence and direction of fans and wind and check, if an appreciable air draft or turbulence is being created.

**K-1.2.3** Explain the sampling procedure, purpose of sampling and how it may be most helpful to the worker.

**K-1.2.4** The sampling time should be so adjusted that it is possible to collect the sample for desired duration without interruptions.

**K-1.2.5** Attach sampler to the worker with the help of a waist belt. The filter holder shall be in the breathing zone of the worker.

NOTE — It is a good practice to attach the filter holder always at the same place in the breathing zone.

**K-1.2.6** The worker should be comfortable with sampler and should be able to perform his routine duties without any discomfort.

**K-1.2.7** Remove the plastic cap from the face of the filter, switch on the pump and note the time with the help of a stop watch.

**K-1.2.8** Remain in a position to watch any abnormal activity of the worker or in surroundings, which may influence results.

**K-1.2.9** Check in between, whenever possible, that the pump is running.

**K-1.2.10** After the scheduled time switch off the pump and record the duration of sampling.

**K-1.2.11** Check the flowrate after sampling has been completed.

**K-1.2.12** Replace the plastic cap and remove the filter holder.

**K-1.3 Processing of Sample**

**K-1.3.1** Clean the work bench, and place a clean sheet of paper.

**K-1.3.2** Clean and check all instruments which may be required for processing.

**K-1.3.3** Clean microscope glass slides and cover slips, and carefully label the slides.

**K-1.3.4** Check that adequate amount of acetone is present in acetone apparatus and water is running through condenser column.

**K-1.3.5** Wipe of the vapour outlet for any liquid acetone. Check that vapours are being formed and escape through outlet in adequate quantity.

**K-1.3.6** Remove caps from filter holder and gently take out the filter with the help of a special forceps, supplied with the kit. Never use a pointed forcep.

**K-1.3.7** Check filter for any evidence of tampering or damage.

**K-1.3.8** Divide the filter with the help of scalped, by rolling action in case required.

**K-1.3.9** Place filter piece on glass slide, dust side up.

**K-1.3.10** Keep slide with filter near the outlet at a distance of 15 to 25 mm and release acetone vapours for 3 - 5 s, taking care that no liquid droplets fall on the filter.

**K-1.3.11** Ensure that filter is made completely transparent and there is no distortion of filter.

**K-1.3.12** Place 2-3 drops of triacetin on the slide with the help of a syringe attached with a filtering mechanism.

**K-1.3.13** Take a clean cover slip and gently lower it down immediately, and ensure that no air bubbles have formed.

**K-1.3.14** Leave slides overnight for clearing. Alternatively, the slides may be placed in an oven for 15 min at  $50 \pm 2^\circ\text{C}$  to hasten clearing but this practice should be avoided as far as possible.

NOTE — It is important that all laboratory work is carried out in a dust free environment.

## **K-1.4 Counting and Sizing of Fibres**

**K-1.4.1** Clean microscope eyepieces, objective lens and substage with lens cleaning fluid or xylene.

**K-1.4.2** Check that the phase rings of microscope are properly aligned.

**K-1.4.3** Check resolution power of microscope with HSE/NPL detection limit test slide Mark II (*see* Annex E).

**K-1.4.4** Count and size fibres strictly according to 4.3.4 and Annex L.

**K-1.4.5** Make use of impressions given in Walton-Backett Graticule, as much as possible for counting and sizing of fibres.

**K-1.4.6** Record all relevant information in data sheet given in Annex C.

**K-1.4.7** While presenting results enclose all the relevant information on sampling for proper interpretation of results.

## **ANNEX L**

( *Clause* K-1.4.4 )

### **BASIC GROUPS OF ASBESTOS FIBRES**

**L-1** Asbestos fibres collected on membrane filters fall into four basic groups as detailed in L-1.1 to L-1.4.

#### **L-1.1 Single Fibres (*see* Fig. 8)**

These are the simplest of the fibres to identify and count. They are the most common of measurable fibres as seen on the membrane filter. Chrysotile fibres, while sometimes straight, often assume a curved or curly outline. Fibres which appear irregular and perhaps unfibre-like are counted, if they conform to the basic requirements of fibre definition.

#### **L-1.2 Split Fibres (*see* Fig. 9)**

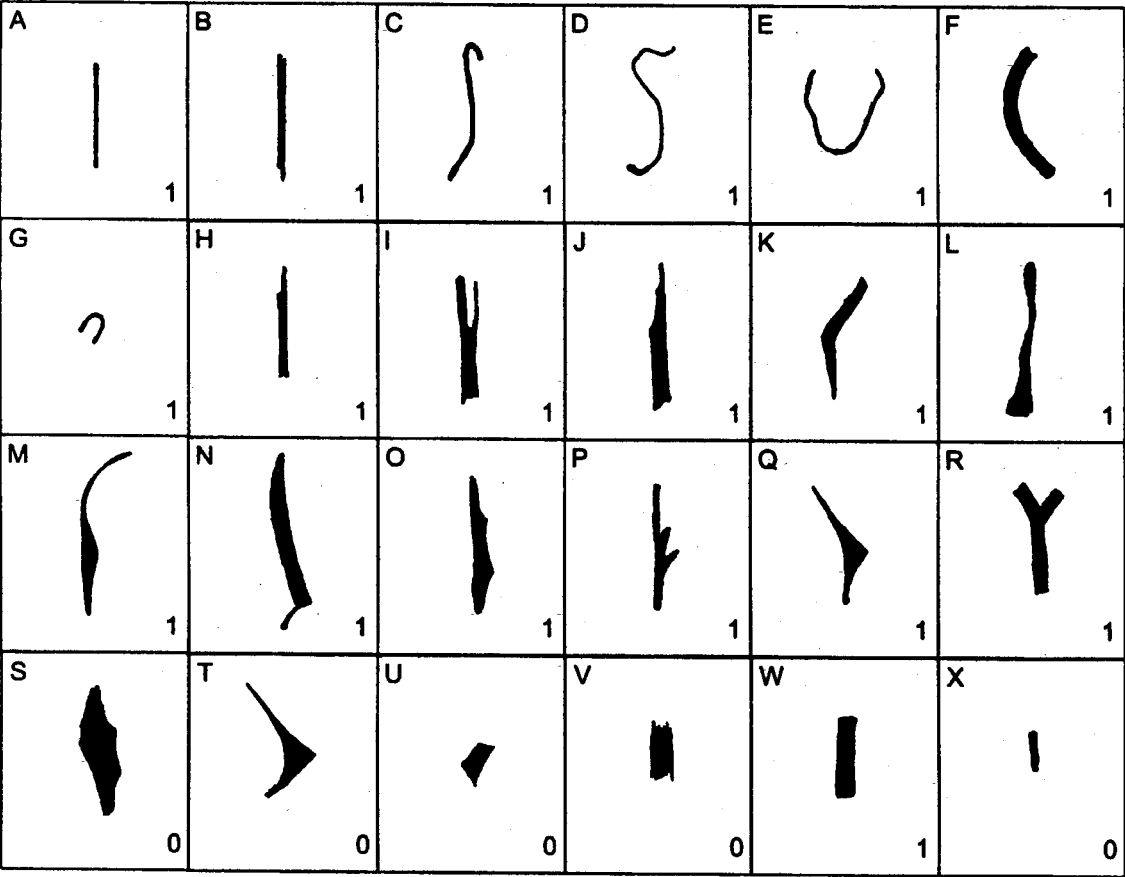
These appear generally as a fibre or fibres splitting away from a single stem.

#### **L-1.3 Grouped Fibres (*see* Fig. 10)**

These are formed when fibres overlap, inter twine or pack together. The simplest form is when two fibres overlap and cross each other. In this case each fibre in the group appears as a discrete entity. In more complex form fibres lie nearly parallel and appear to originate from the same bundle.

#### **L-1.4 Fibres with Other Particles (*see* Fig. 11)**

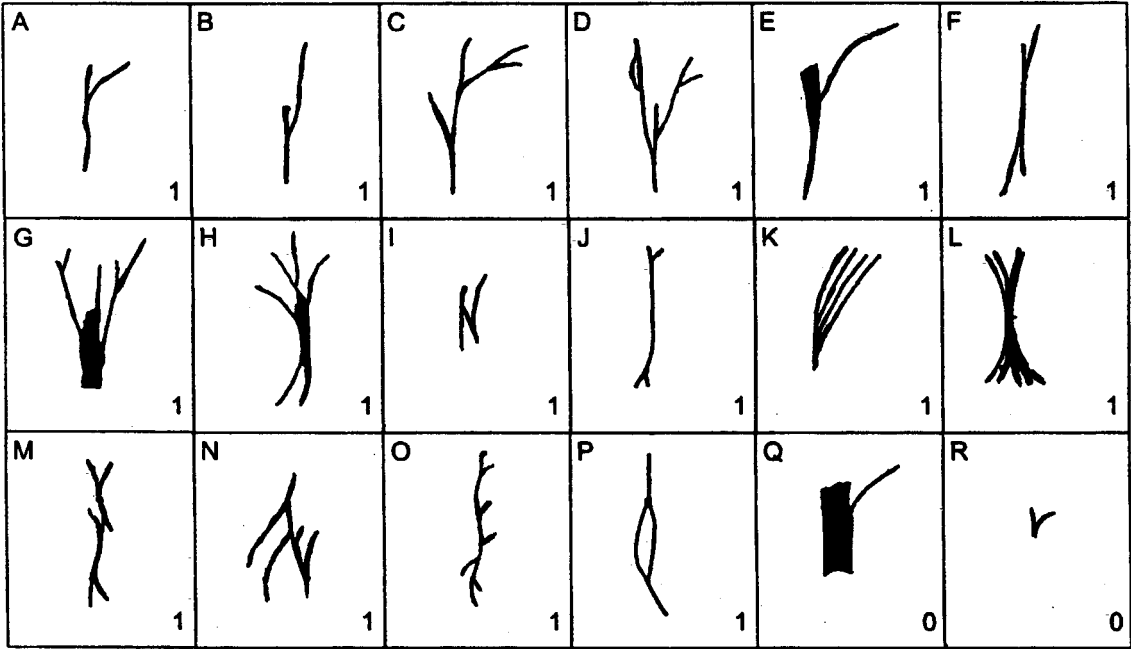
This group consist of fibres attached to, or embedded in particulate matter. This latter material could be parent asbestos rock, or resins, cement, silicates, etc, as used in manufactured product. Under the microscope some fibre, especially chrysotile, appears to project from the particulate matter with only part of the fibre seen. Other fibres are seen as embedded in the particulate matter.



Scale:  
  
5  $\mu$ m

NOTE — The number in the right bottom corner of each drawing indicates the number of fibres counted.

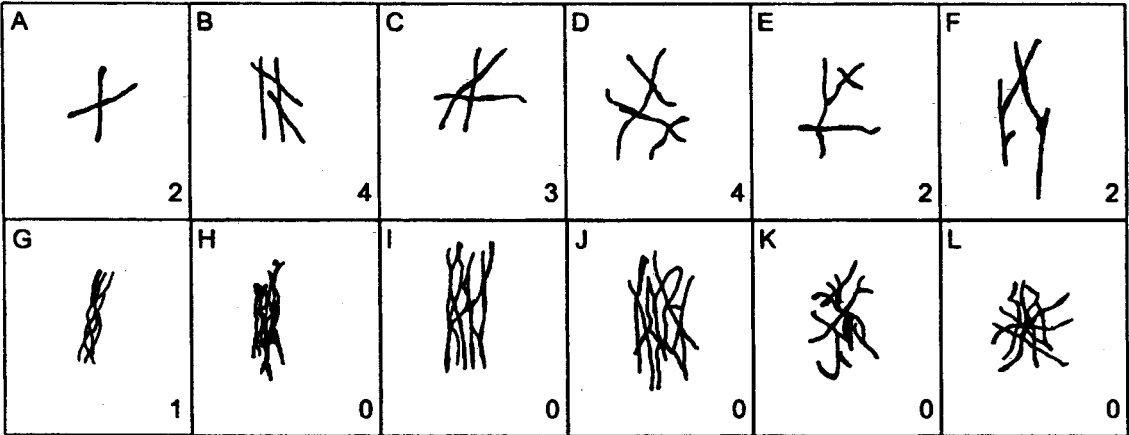
FIG. 8 SINGLE ASBESTOS FIBRE



Scale:  
  
5  $\mu$ m

NOTE — The number in the right bottom corner of each drawing indicates the number of fibres counted.

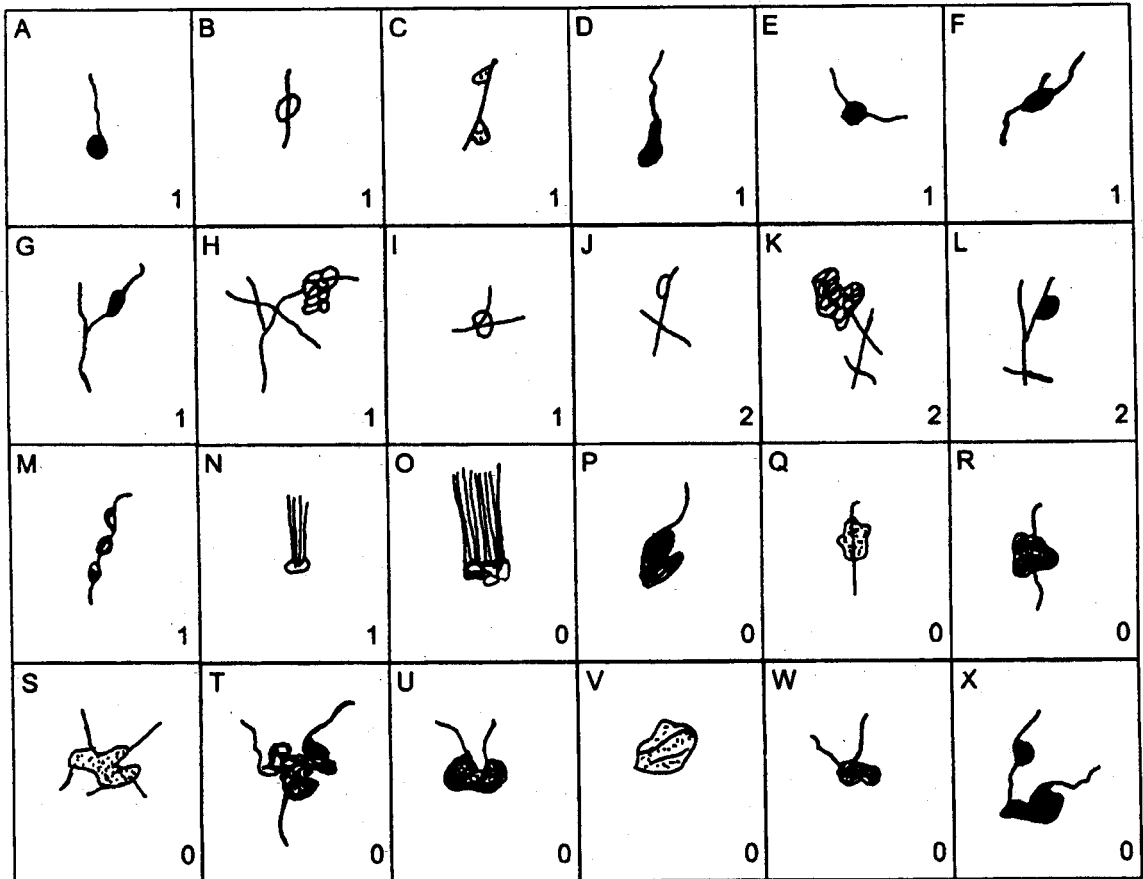
FIG. 9 SPLIT ASBESTOS FIBRE



Scale:  
  
5  $\mu$ m

NOTE — The number in the right bottom corner of each drawing indicates the number of fibres counted.

FIG. 10 GROUPED ASBESTOS FIBRE



Scale:

  
5  $\mu$ m

NOTE — The number in the right bottom corner of each drawing indicates the number of fibres counted.

FIG. 11 ASBESTOS FIBRES WITH OTHER PARTICLES

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